



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES

Applicant : Kambe et al.
Serial No.: 08/962,362
Filed : October 31, 1997
For : PHOSPHORS
Docket No.: N19.12-0006

Appeal No.

Group Art Unit: 2879

Examiner: M. Day

AFM
2879
#23
Appeal
Brief
J. McMillan
11/14/00

**TRANSMITTAL OF APPEAL BRIEF
(PATENT APPLICATION - 37 CFR § 192)**

Commissioner of Patents and Trademarks
Washington, D.C. 20231

I HEREBY CERTIFY THAT THIS PAPER IS
BEING SENT BY U.S. MAIL, FIRST
CLASS, TO THE COMMISSIONER OF
PATENTS AND TRADEMARKS, WASHINGTON,
D.C. 20231. THIS

25 DAY OF October, 2000
Robert J. Ward
PATENT ATTORNEY

Sir:

Transmitted herewith in triplicate is the Appeal Brief in
this application with respect to the Notice of Appeal filed on July
25, 2000.

FEE STATUS

[X] Small entity status under 37 CFR §§ 1.9 and 1.27 is
established by a verified statement.

FEE FOR FILING APPEAL BRIEF

Pursuant to 37 CFR 1.17(c) the fee for filing the Appeal
Brief is \$150.00.

The Commissioner is authorized to charge any additional
fees associated with this paper or credit any overpayment to
Deposit Account No. 23-1123. A duplicate copy of this

communication is enclosed.

Respectfully submitted,

WESTMAN, CHAMPLIN & KELLY, P.A.

By: Peter S. Dardi
Peter S. Dardi, Ph.D., Reg. No. 39,650
Suite 1600 - International Centre
900 Second Avenue South
Minneapolis, Minnesota 55402-3319
Phone: (612) 334-3222 Fax: (612) 334-3312

PSD:nhw



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BOX AF
Assistant Commissioner For Patents
Washington, D.C. 20231

I HEREBY CERTIFY THAT THIS PAPER IS
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COMMISSIONER FOR PATENTS,
WASHINGTON, D.C. 20231. THIS

25 DAY OF October, 2000.

Robert S. Dardi
PATENT ATTORNEY

Sir:

This is an appeal from an Office Action dated April 25, 2000 in which claims 1-6 and 20-30 were finally rejected. A Notice of Appeal was filed on July 25, 2000. A Petition for Extension of Time for a one month extension was filed on October 10, 2000. Applicants believe that no additional extensions of time are needed. If additional extensions of time are needed, consider this paper as a petition for such an extension.

REAL PARTY IN INTEREST

NanoGram Corporation, a corporation organized under the laws of the state of Delaware, and having offices at 46774 Lakeview Boulevard, Fremont, California, has acquired the entire right, title and interest in and to the invention, the application, and any and all patents to be obtained therefor, as set forth in the Assignment filed with the patent application and recorded on Reel 9091, frame 0039.

RELATED APPEALS AND INTERFERENCES

There are no related appeals or interferences regarding the present appeal.

STATUS OF THE CLAIMS

Claims 1-6 and 20-30 stand rejected. The pending claims are listed in Appendix A.

STATUS OF AMENDMENTS

All Amendments have been entered. Examiner Day indicated by phone on October 19, 2000 that a Declaration submitted with an Amendment After Final on October 10, 2000 was not considered as untimely. This Declaration by Professor Bricker is submitted with this Appeal Brief.

SUMMARY OF INVENTION

Applicants' invention involves display devices incorporating highly uniform fluorescent particles. The fluorescent particles emit light in response to stimulation, such as electrical stimulation. Therefore, the particles are suitable for the incorporation into display devices wherein the particles are selectively excited to produce an image. A variety of suitable display devices and corresponding structures are described in Applicants' specification at page 22, line 4 to page 24, line 24.

The use of highly uniform particles provides for greater control over the emissions of the particles. See page 19, line 21 to page 20, line 4. Specifically, particles with a narrow particle size distribution have a corresponding light emission band covering a narrow frequency range. See page 4, lines 25-29 and page 19, line 30 to page 20, line 4. In addition, the uniformity of the particles leads to processing advantages with respect to the formation of thin layers with sharp edges. See page 20, lines 5-13. The production of highly uniform nanoparticles is enabled by

the laser pyrolysis approach described in Applicants' specification.

Laser pyrolysis for particle generation involves a reactant stream that flows through an intense light beam, such as a laser beam. See, for example, page 5, lines 16-26. The light beam drives the chemical reaction. See, for example, page 7, line 24 to page 8, line 5. Because of the intense temperatures generated by absorption of light by compounds flowing through the light beam, the light beam forms a well defined reaction zone. See page 8, lines 14-15. The particles are quenched rapidly after leaving the reaction zone. See, for example, page 11, lines 16-18. Having a very well defined reaction zone results in the extreme uniformity of the resulting particles. See, for example, page 11, lines 18-20.

The resulting particles produced by laser pyrolysis have a very narrow particle size distribution. In particular, the particles have a narrow distribution about the average particles size, as determined by transmission electron microscopy. See, for example, page 19, lines 4-16. Transmission electron micrographs of titanium oxide formed by laser pyrolysis from a copending patent application are enclosed in Appendix C, as visual confirmation of particle uniformity.

These highly uniform particles are particularly well suited for the formation of improved display devices. See, for example, page 4, lines 29-32. Sharp edges can be formed between elements of the display. See, for example, page 20, lines 8-13. The small particles are also suitable for low-velocity electronic excitation with high luminosity. See, for example, page 21, lines 29-32.

ISSUES

1. Whether claims 1, 4-6, 20-25 and 27-30 are obvious over U.S. Patent 5,442,254 to Jaskie et al.?
2. Whether claims 2, 3 and 26 are obvious over U.S. Patent 5,442,254 to Jaskie et al. in view of U.S. Patent 5,455,489 to Bhargava?

GROUPING OF CLAIMS

Claims 1-6 and 20-30 are within a single claim group.

ARGUMENT

1. Rejections Under 35 U.S.C. §103(a) Over Jaskie

The Examiner rejected claims 1, 4-6, 20-25 and 27-30 under 35 U.S.C. §103(a) as being unpatentable over U.S. Patent 5,442,254 to Jaskie (the Jaskie patent). A copy of the Jaskie patent is found in Appendix C. As noted by the Examiner, the Jaskie patent discloses a display device including fluorescent particles and the desirability of having highly uniform fluorescent particles. The Jaskie patent further describes average particle sizes overlapping with the present average particle size range specified in Applicants' claims. The Jaskie patent also discloses the concept of tuning fluorescent emissions by selecting the particle size.

The Examiner noted that the Jaskie patent did not specifically describe the range of particle sizes. It was the Examiner's position that the production of particles having the claimed particle size distribution was within the skill in the art. Based on this asserted level of skill in the art, the Examiner indicated that the claimed narrow particle size distribution was obvious. The Examiner further indicated that the Jaskie patent taught that the specification of a desired particle size range was within the skill in the art. See the Jaskie patent at column 7, lines 34-40.

In response to the Examiner's assertions, Applicants argued that the production of particles with the claimed narrow particle size distribution was not within the level of skill in the art. Furthermore, Applicants presented evidence that the discussion in the Jaskie patent did not permit the formation of the narrow particle size distributions disclosed and claimed by Applicants. Thus, Applicants believe that this evidence has rebutted assertions of obviousness.

As objective evidence, Applicants submitted a Declaration by Professor Singh, an expert in nanoparticle technology, with a Preliminary Amendment dated March 24, 2000. A copy of Professor Singh's Declaration is found in Appendix D. Professor Singh's Declaration supports arguments that the Jaskie patent did not enable the production of Applicants' claimed invention. The Examiner did not find the Singh reference convincing. Below, Applicants present further arguments for patentability including a Declaration by Professor Bricker, assertions regarding the persuasiveness of Dr. Singh's Declaration, and additional objective evidence regarding patentability over the Jaskie reference. The aggregate evidence is the basis for further arguments below that the record as a whole does not support a finding of obviousness.

A. Legal Background

The proposition is well established that the prior art only renders a composition of matter or apparatus unpatentable to the extent that the prior art provides a means of obtaining the composition or apparatus.

To the extent that anyone may draw an inference from the Von Bramer case that the mere printed conception or the mere printed contemplation which constitutes the designation of a 'compound' is sufficient to show that such a compound is old, regardless of whether the compound is involved in a 35 U.S.C. 102 or 35 U.S.C. 103 rejection, we totally disagree. ... We think, rather, that the true test of any prior art relied upon to show or suggest that a chemical compound is

old, is whether the prior art is such as to place the disclosed 'compound' in the possession of the public.

In re Brown, 141 USPQ 245, 248-49 (CCPA 1964) (emphasis in original) (citations omitted). Similarly, see In re Hoeksema, 158 USPQ 596, 600 (CCPA 1968) (emphasis in original):

We are certain, however, that the invention as a whole is the claimed compound and a way to produce it, wherefore appellant's argument has substance. There has been no showing by the Patent Office in this record that the claimed compound can exist because there is no showing of a known or obvious way to manufacture it; hence, it seems to us that the 'invention as a whole,' which section 103 demands that we consider, is not obvious from the prior art of record.

While there are valid reasons based on public policy as to why this defect in the prior art precludes a finding of obviousness under section 103, In re Brown, supra, its immediate significance in the present inquiry is that it poses yet another difference between the claimed invention and the prior art which must be considered in the context of section 103. So considered, we think the differences between appellant's invention as a whole and the prior art are such that the claimed invention would not be obvious within the contemplation of 35 U.S.C. 103.

The Federal Circuit has further emphasized these issues.

"But to be prior art under section 102(b), a reference must be enabling. That is, it must put the claimed invention in the hands of one skilled in the art." In re Sun, 31 USPQ2d 1451, 1453 (Fed. Cir. 1993) (unpublished). Assertions in a prior art reference do not support an anticipation or obviousness rejection unless the references place the claimed invention in the hands of the public. Beckman Instruments Inc. v. LKB Produkter AB, 13 USPQ2d 1301, 1304 (Fed. Cir. 1989). "In order to render a claimed apparatus or method obvious, the prior art must enable one skilled in the art to make and use the apparatus or method." Id. While a reference is prior art for all that it teaches, references along with the knowledge of a person of ordinary skill in the art must be enabling to place the invention in the hands of the public. In re Paulsen,

31 USPQ2d 1671, 1675 (Fed. Cir. 1994). See also In re Donohue, 226 USPQ 619, 621 (Fed. Cir. 1985).

In evaluating obviousness, the level of skill in the art must be considered. MPEP §2141, citing Graham v. John Deere, 383 U.S. 1, 148 USPQ 459 (1966). In the present case, a person of ordinary skill in the art would have, at least, a bachelors degree in electrical engineering, material science or physics, and with experience in particle technology and/or the fluorescent properties of material. As will be discussed in detail below, the technology discussed in the Jaskie Patent for particle separation is unconventional in the technology area. Therefore, we can look to persons of skill in the separation technology to evaluate the disclosure of wet filtration in the Jaskie patent. However, a person of ordinary skill in the art of fluorescent nanoparticles would be a person with skill in inorganic material science, electrical engineering or physics and would have limited understanding of chromatographic separation technology. Therefore, Applicants believe that Professor Bricker would have significant extraordinary skill in separation technology relative to a person of ordinary skill in the **relevant** technology.

Under a factual inquiry relating to an obviousness analysis, objective evidence **must** be considered. See, MPEP §2141, and Graham v. John Deere, 383 U.S. 1, 148 USPQ 459 (1966). Applicants present two Declarations Under 37 C.F.R. §1.132. Responding to Professor Singh's Declaration in the Office Action of April 25, 2000, the Examiner stated that "To be of probative value, any objective evidence should be supported by actual proof." MPEP 716.01(c), however, indicates that "[opinion] testimony is entitled to consideration and some weight so long as the opinion is not on the ultimate legal conclusion." "In assessing the probative value of an expert opinion, the examiner **must** consider the nature of the matter sought to be established, the strength of any opposing evidence, the interest of the expert in the outcome of the case,

and the presence and absence of factual support for the expert's opinion." Id. (Emphasis added). These statements in the MPEP are consistent with the statements of the Supreme Court in Graham v. John Deere.

"[T]he conclusion of obviousness vel non is based on the preponderance of evidence and argument in the record." In re Oetiker, 24 USPQ2d 1443, 1445 (Fed. Cir. 1992). The patent office has the ultimate burden of persuasion in establishing that an applicant is not entitled to a patent. Id. at 1447, concurring opinion of Judge Plager. **"The only determinative issue is whether the record as a whole supports the legal conclusion that the invention would have been obvious."** Id.

B. Declaration by Professor Bricker

A Declaration by Professor Bricker under 37 C.F.R. §1.132 is enclosed with this Appeal Brief in Appendix E. Applicants originally submitted this Declaration with an Amendment after Final of October 10, 2000. The Examiner considered the Declaration untimely and did not consider the Declaration.

The Jaskie patent includes a description of a "wet filtering technique" at column 7, lines 28-40. This wet filtration involves the formation of a suspension of the particles and the use of a cloth put into the suspension to draw up the particles. Professor Bricker's Declaration is specifically directed to extreme deficiencies of the wet filtration approach described in the Jaskie patent. Professor Bricker's Declaration also describes why other chromatographic techniques, including well established methods, would not be expected to accomplish the extremely fine separation needed to obtain the claimed invention.

Professor Bricker is an expert in separation technologies similar to the "wet filtration" approaches described in the Jaskie patent at column 7, lines 28-40. These separation techniques were developed for the separation of chemical species. In addition, these techniques have been generalized for the separation of

biological macromolecules, which have a nanometer size scale. Dr. Bricker's Declaration presents an explicit and clear explanation of why the process described in the Jaskie patent cannot and will not work for the intended purpose. Thus, **Applicants have presented clear objective evidence that the Jaskie patent does not enable the production of Applicants' claimed invention.** Applicants note that Professor Bricker has no interest in the outcome of the present application.

C. Declaration by Professor Singh

Applicants filed an Declaration under 37 C.F.R. §1.132 by Professor Singh with a Preliminary Amendment on March 24, 2000. A copy of Professor Singh's Declaration is presented in Appendix D. In response to Dr. Singh's Declaration submitted by Applicants, the Examiner has indicated that the Declaration had "little probative value." See the Office Action of April 25, 2000 at page 5. The Examiner further indicated that objective evidence "should be supported by actual proof." The Examiner cited for support MPEP 716.01(c). Applicants respectfully assert that the Examiner incorrectly cited the MPEP and the underlying case law. The law is summarized above. In addition, the factors described by the MPEP for evaluating the weight of a Declaration are summarized above.

In view of establishing the weight to be accorded to Professor Singh's Declaration, Applicants note that Professor Singh has no interest to be gained in the present case. Dr. Singh is an expert who has consulted with many important companies in the field of nanotechnology. As noted in the Declaration, Dr. Singh has no equity interest in NanoGram. Any expert will require payment for their time. He is not an inventor and has not consulted for NanoGram in the area of phosphors, except for the Declaration under discussion. Dr. Singh has **no interest** in the outcome of the present patent application.

The Examiner indicated that Dr. Singh's Declaration was self-contradictory because of statements relating to reasons why

chromatographic techniques have not been explored for the separation of inorganic nanoparticles. Applicants firmly believe that Dr. Singh's Declaration is not self-contradictory. Dr. Singh was addressing a speculative proposition, the separation of nanoparticles by size using chromatography, i.e., wet filtration in the terminology of the Jaskie patent. To state that the procedure would be difficult or impossible to scale up is one explanation of why no work has been reported on the approach or related approaches to date. Since no work had been done previously, an undue amount of experimentation would be required to attempt to practice the technique. This argumentation is **completely self-consistent**.

Applicants do not deny that generally chromatography is a well developed field for **chemical and biochemical separation**. However, this experience does not extend to the separation of solid inorganic particles. The Examiner cited isotope separation of lithium in 1938, as described in Instruments of Science, An Historical Encyclopedia (Garland Publishing, Inc.). A copy was not forwarded with the Office Action. Applicants thank the Examiner for subsequently providing a copy of the article. As described in the reference on page 108, the lithium isotope separation was performed by ion-exchange chromatography. Ion exchange chromatography is performed to separate molecular ions or atomic ions in solution. Thus, ${}^6\text{Li}^+$ and ${}^7\text{Li}^+$ have slightly different equilibrium constant with respect to adsorption on the ion-exchange resin. These solvated ions have no direct relationship to inorganic nanoparticles, and they are separated by **mass not by size**. The lithium isotopes differ only by the number of neutrons that varies the mass and have an identical atomic size.

Since ion exchange is used for **solvated molecular or atomic ions**, there is no expectation that ion-exchange chromatography would be expected to work for inorganic nanoparticles to separate them by size. Professor Bricker's Declaration directly addressed the application of chromatography to

the size separation of nanoparticles with respect to describing why these methods would not be expected to work. Therefore, Applicants do not believe that there is any evidence whatsoever contrary to their position and the expert opinion of Professor Singh that, minimally, an undue amount of experimentation would be required to attempt to separate nanoparticles by size using wet filtration described in the Jaskie patent or for that matter any type of chromatographic technique. The evidence of record strongly suggests that such a chromatographic separation of nanoparticles based on size would be actually impossible based on the present state of technology.

In the present case, the nature of the matter sought to be established, i.e., size separation of nanoparticles by chromatography, is at best speculative. It is difficult to establish that a method that has never been tried is not a useful approach. To establish a new method of purifying nanoparticles based on the minimal guidance from the Jaskie patent is at most an invitation to perform extensive research in the hopes that it may work. Professor Singh's Declaration addressed the relevant issues from the perspective of an **expert** in the field of inorganic particles regarding the suggestions in the Jaskie patent.

Nevertheless, to confirm that Dr. Singh's expert statements were well founded, Applicants have obtained a Declaration from an expert in chromatography, Dr. Bricker, who has directly addressed the disclosure in the Jaskie patent. Professor Bricker concluded that the Jaskie "wet filtration" will not work to separate nanoparticles. Dr. Bricker's expert Declaration has presented objective evidence to directly address the issues raised in the Jaskie patent. The conclusions that follow from Dr. Bricker's analysis are consistent with and support Dr. Singh's statements. Dr. Bricker was not aware of Dr. Singh's Declaration. In addition, Applicants present below additional objective evidence that the best conventional filtering approaches available for

nanoparticles are not sufficient to perform the necessary particle separation to practice Applicants' claimed invention.

In summary, there is no evidence, which can withstand scrutiny, contrary to Dr. Singh's statements regarding the disclosure in the Jaskie patent. The Jaskie patent does not present any experimental results. Almost five years after the Jaskie patent issued, there is no public knowledge of successful application of the Jaskie approach. This failure regarding the practice of the Jaskie invention is objective evidence against the Jaskie suggestion. Applicants have further supported the opinions in Dr. Singh's Declaration by a Declaration by Dr. Bricker, an expert in separation technologies, and by objective evidence, below, regarding the lack of availability of commercial separation approaches suitable to performed the specified particle separations.

Together, the Declarations by Professor Singh and Professor Bricker provide overwhelming objective evidence from a person with considerable experience in the separation of biological nanoparticles as well as the perspective of an expert in inorganic nanoparticle technology that the approach discussed in the Jaskie patent will not work produce the compositions disclosed and claimed by Applicants.

D. Patentability Over the Jaskie Patent

The Jaskie patent makes vague references to approaches to size separate inorganic nanoparticles. In particular, the Jaskie patent discusses wet filtration as an approach for separating inorganic nanoparticles. Presumably, the vague Jaskie reference to wet filtration is based on a desire to adapt methods used for biological macromolecules for the separation of inorganic nanoparticles.

It follows from Dr. Bricker's Declaration that the description of wet filtration in the Jaskie patent is not well conceived. In particular, the Jaskie patent provides virtually no

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guidance in the materials suitable to perform these miraculous separations. The Jaskie patent specifically refers to the use of cloth. Perhaps this reference to cloth is an inadvertent mistake to a reference to paper chromatography. According to the Instruments of Science article at page 108 supplied by the Examiner, paper chromatography is performed with "specially made filter paper and airtight development tanks containing the solvent." The Jaskie article seems to indicate that cloth is a substitute for the specially made filter paper, and no reference is made to the solvent. The deficiencies of the Jaskie process are clearly and thoroughly described in Professor Bricker's Declaration.

Applicants have presented objective evidence in the form of Declarations of Dr. Bricker, an expert in separation technology, and Dr. Singh, an expert in inorganic nanoparticle technology, that the "wet filtration" approaches disclosed in the Jaskie patent at column 7, lines 28-40 will not work to obtain particles with a selected narrow particle size. These Declarations provide further evidence that the production of highly uniform nanoparticles, as claimed by Applicants were not within the level of ordinary skill in the art. Specifically, Professor Bricker's Declaration indicates that other forms of chromatography would also not work, and Professor Singh's Declaration indicates that appropriate separation technologies were not known to a person of skill in the inorganic nanoparticle art. These assertions are consistent with the deficiencies in the Jaskie patent since Jaskie is clearly a person of high skill in the art of fluorescent nanoparticles. Since Jaskie in the Jaskie patent could not describe how to obtain the desired nanoparticles, presumably a person of ordinary skill in the inorganic nanoparticle art would not have skill required to perform the desired separation.

As further evidence that the particle separation was not within the skill in the art, Applicants present objective data in the form of product information from a commercial supplier of state

of the art inorganic particle filtration technology. In particular, Applicants have enclosed information in Appendix F downloaded from the Millipore Corporation (Millipore) web site. Millipore is a leader in filtration technology. Uniformity of particles is also a desirable feature for inorganic particles used in the fine polishing of electronic substrates, generally referred to chemical-mechanical polishing or CMP. This information from the Millipore website indicates that in 1999 standard CMP slurries have a significant fraction of larger particles combined with the desired nanoscale particles.

A plot from Millipore of the removal capability of Millipore's line of Planargard™ filters used to filter surface polishing slurries is included in the enclosed materials. These filters are not perfectly effective for removing particles even with diameters of greater than one micron. In addition, the filters are not effective for distinguishing a cut-off of particles less than a micron. Applicants' claims indicate that the average particle size is less than 100 nm. Thus, filtration using state of the art commercial filters is not an effective means of creating the narrow particle size distributions, as disclosed and claimed by Applicants.

In contrast with the Jaskie approach of wet filtration, Applicants' particle production approach forms a narrow distribution of particle sizes during the formation of the particles. The average particle size can be adjusted by changing the reaction parameters. Thus, no separation of the particles is needed. Since the Jaskie patent does not place the public in possession of Applicants' claimed invention, the Jaskie patent does not render Applicants' claimed invention obvious. **Applicants believe that they have more than met their burden in overcoming prima facie obviousness.**

The weight of evidence viewed in its entirety does not support a finding of unpatentability. Specifically, the Patent

Office has not met their burden of persuasion with respect to unpatentability. Applicants respectfully request the withdrawal of the rejection of claims 1, 4-6, 20-25 and 27-30 under 35 U.S.C. §103(a) as being unpatentable over the Jaskie patent.

2. Rejections Over Jaskie and Bhargava

The Examiner rejected claims 2, 3 and 26 under 35 U.S.C. §103(a) as being unpatentable over the Jaskie patent in view of U.S. Patent 5,455,489 to Bhargava (the Bhargava patent). The Examiner cited the Jaskie patent for the reasons described with respect to claim 1. The Examiner cited the Bhargava patent for disclosing ZnO, ZnS and Y₂O₃ as phosphors. The Examiner further cited the Bhargava patent for disclosing the use of phosphors in an electroluminescent display. Applicants respectfully request reconsideration of the rejections based on the following comments.

As described in detail above, the Jaskie patent is deficient with respect to disclosing Applicants' claimed invention based on a narrow particle size distribution. The Bhargava patent does not teach or suggest highly uniform phosphor particles, as disclosed and claimed by Applicants. Thus, the Bhargava patent does not make up for the deficiencies of the Jaskie patent.

Since the Jaskie patent and the Bhargava patent do not teach appropriate approaches to produce phosphor particles with the narrow particle size distribution disclosed and claimed by Applicants, the combined disclosures of the two patents do not render Applicants' claimed invention obvious. Applicants respectfully request the withdrawal of the rejection of claims 2, 3 and 26 under 35 U.S.C. §103(a) as being unpatentable over the Jaskie patent in view of the Bhargava patent.

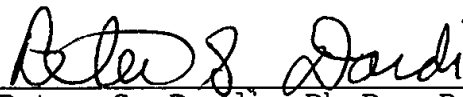
CONCLUSIONS

Applicants submit that claims 1-6 and 20-30 are unobvious over the prior art of record. Applicants believe that the Patent

Office has failed to meet their burden of persuasion with respect to unpatentability of any of the claims in view of the total evidence presented. Thus, Applicants respectfully request the reversal of the rejections of claims 1-6 and 20-30 and the allowance of claims 1-6 and 20-30.

Respectfully submitted,

WESTMAN, CHAMPLIN & KELLY, P.A.

By: 

Peter S. Dardi, Ph.D., Reg. No. 39,650
Suite 1600 - International Centre
900 Second Avenue South
Minneapolis, Minnesota 55402-3319
Phone: (612) 334-3222 Fax: (612) 334-3312

PSD:nhw



Appendix A

PENDING CLAIMS

1. A display device comprising phosphor particles having an average diameter less than about 100 nm and wherein the phosphor particles comprise a collection of particles having a diameter distribution such that at least about 95 percent of the particles have a diameter greater than about 40 percent of the average diameter and less than about 160 percent of the average diameter.
2. The display device of claim 1 wherein the phosphor particles comprise a metal compound selected from the group consisting of ZnO, ZnS, TiO₂ and Y₂O₃.
3. The display device of claim 2 wherein the metal compound is ZnO.
4. The display device of claim 1 wherein the phosphor particles have an average diameter from about 5 nm to about 50 nm.
5. The display device of claim 1 wherein the collection of particles have a diameter distribution such that at least about 95 percent of the particles have a diameter greater than about 60 percent of the average diameter and less than about 140 percent of the average diameter.
6. The display device of claim 1 wherein the light emission follows low velocity electron excitation.
20. The display device of claim 1 wherein the phosphor particles further comprise a second collection of particles, the second collection of particles having a diameter distribution such that at least about 95 percent of the particles have a diameter

greater than about 40 percent of the average diameter and less than about 160 percent of the average diameter.

21. The display device of claim 1 wherein the phosphor particles are in contact with an anode.

22. The display device of claim 1 further comprising a liquid crystal layer.

23. The display device of claim 1 further comprising a partially light transparent substrate.

24. The display device of claim 1 further comprising a transparent electrode comprising indium tin oxide.

25. The display device of claim 1 further comprising an electrode to guide the electrons from the cathode to the anode.

26. The display device of claim 1 wherein the display is an electroluminescent display.

27. The display device of claim 1 wherein the device is a field emission device with the phosphor particles located between an anode and cathode.

28. The display device of claim 27 comprising a plurality of anodes and cathodes where each electrode pair forms an addressable pixel.

29. The display device of claim 1 wherein the phosphor particles are roughly spherical.

30. The display device of claim 1 wherein the phosphor particles are excitable by low velocity electrons.



Appendix B

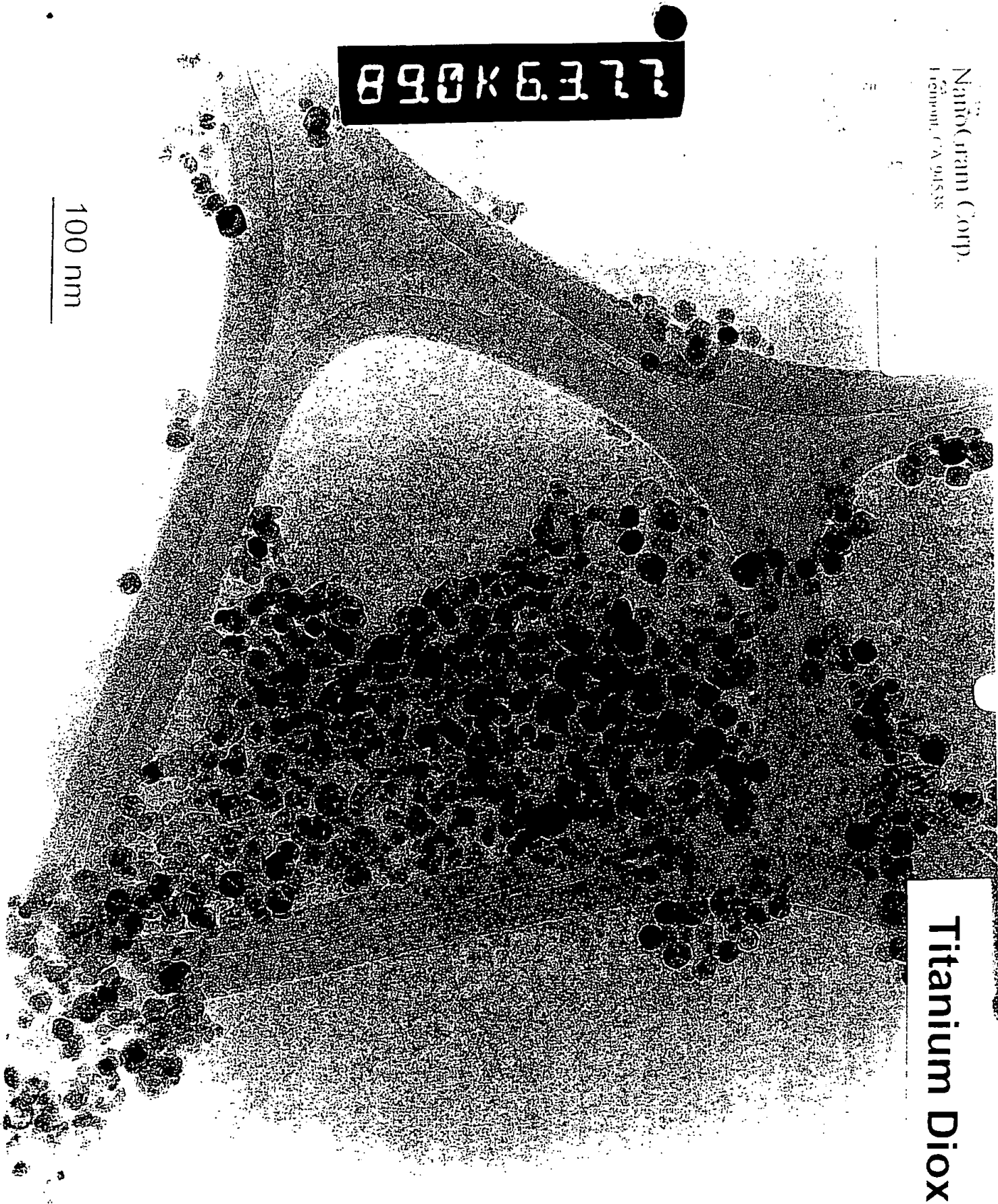
TRANSMISSION ELECTRON MICROGRAPH OF TITANIUM OXIDE
FORMED BY LASER PYROLYSIS

NanoGum Corp.
Fremont, CA 94538

890K6377

Titanium Dioxide

100 nm





Appendix C

Cited References

1. U.S. Patent 5,442,254 to Jaskie
2. U.S. Patent 5,455,489 to Bhargava

Appendix D

DECLARATION UNDER 37 C.F.R. §1.132
OF
PROFESSOR RAGIV SINGH

Resume of Dr. Singh is also attached.



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Kambe et al.

Serial No.: 08/962,362

Filed : October 31, 1997

For : PHOSPHORS

Docket No.: N19.12-0006

Group Art Unit:
2879

Examiner: M. Day

DECLARATION UNDER 37 C.F.R. § 1.132

Express Mail: EL418983858US

Date of Deposit: March 24, 2000

Assistant Commissioner for Patents
Washington, D.C. 20231

I, Rajiv K. Singh, Ph.D., hereby declare as follows:

1. I am presently a Professor of Material Science and Engineering at the University of Florida at Gainesville. Also, I am also Director of the Characterization, Research Instrumentation and Testbed Facility of the Engineering Research Center for Particle Science and Technology at the University of Florida. Apart from my academic responsibilities, I provide consulting services through R. K. Singh Consulting Inc.
2. I received my Ph.D. degree in 1989 in Material Science and Engineering from North Carolina State University, Raleigh, NC.
3. I have been on the faculty at the University of Florida since 1990. I was promoted to Associate Professor with tenure in 1995 and to full Professor in 1997. A copy of my Curriculum Vitae is attached.
4. My recent accomplishments include receiving a National Science Foundation Young Investigator Award in 1994 and the Hardy Gold Metal for Outstanding Contributions in Material Science in 1995. I was a Distinguished Visiting Professor/Scientist at National University of Singapore (1999) and National Institute for Materials and Chemical Research, Tsukuba, Japan (2000). I am the

author or co-author of more than 293 scientific articles and conference proceedings. I have co-edited five books and guest edited five journal issues.

5. I am under a Consulting Agreement with NanoGram Corporation to provide consulting services in the area of chemical-mechanical planarization. I am not a shareholder in NanoGram Corporation.

6. I have read carefully the pending claims of the above noted patent application entitled "PHOSPHORS" and U.S. Patent 5,442,254 to Jaskie (the Jaskie patent). I did not participate in any capacity with the preparation of the PHOSPHORS patent application.

7. I am very familiar with approaches that have been attempted for separating nanoparticles by filtration. To my knowledge, no experimental results based on the separation of nanoparticles by the wet filtration approaches described in the Jaskie patent at column 7, lines 28-40 have ever been reported in the public literature. Since such chromatography techniques are not known for the separation of nanoparticles, a person of skill in the art would expect to expend a substantial amount of inventive effort to attempt to practice the wet filtration techniques. Without at least some preliminary results to support the basic principles underlying the effort, there would be no reasonable expectation of eventual success at applying the wet filtration approach. In my opinion, the wet filtration approaches described in the Jaskie patent at column 7, lines 28-40 are highly speculative, and the description in the Jaskie patent does not provide a reasonable expectation of successfully separating a collection of nanoparticles to isolate a particle population with a desired narrow range of particle sizes.

8. Since the Jaskie wet filtration techniques would be difficult or impossible to scale up to commercial quantities, it is unlikely that any effort will ever be spent on developing such approaches. The only possibility for the expenditure of experimental effort on such wet filtration techniques would be to

satisfy academic curiosity. In the biological sciences, gel electrophoresis is an important technique for the characterization of biochemical preparations. However, there are other long established approaches for characterizing nanoparticles.

9. I am aware of considerable amounts of effort expended using conventional size exclusion filtration for the preparation of nanoparticle collections. At the time of filing the PHOSPHORS patent application on October 31, 1997, there were no filtration approaches publicly known that could create a collection of phosphorescent nanoparticles with a very narrow size distribution as disclosed and claimed in the PHOSPHORS patent application. At best, these filtration techniques could only exclude micron scale contaminants.

10. Based on my extensive knowledge in the nanoparticle field, it is my opinion that the "tuning (size selection)" described in the Jaskie patent at column 7, lines 28-30 could not be accomplished based on publicly available filtration methods as of October 31, 1997. Specifically, tuning could not be performed by the wet filtration approach outlined in the Jaskie patent without the expenditure of an undue amount of experimentation, if at all.

11. I declare that all statements made herein that are of my own knowledge are true and that all statements that are made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: March 21 '00

By: Rajiv K. Singh
Rajiv K. Singh, Ph.D.

RAJIV K. SINGH

Materials Science and Engineering

University of Florida

Gainesville, FL 32611-2066

(352) 392 1032

(352) 392 3771 (fax)

email: rsing@mail.mse.ufl.edu**Research Interests:**

Innovative processing of materials; Laser processing; thin films; transient thermal phenomena; superconducting and dielectric (low K and high K) thin films; diamond and related materials, rapid thermal processing of elemental and wide band gap semiconductors, chemical-mechanical planarization, particulate coatings; semiconductor processing; modeling of transient thermal processing; flat panel displays, Angstrom scale advanced materials characterization, oxide thin films & electronics, gallium nitride and diamond crystal growth, nanoparticle synthesis and processing, front and back end semiconductor cleaning, phosphors and flat panel displays, thin film batteries.

Education

Ph.D. Materials Science and Engineering, North Carolina State University, Raleigh, 1989

M.S. Materials Science and Engineering, North Carolina State University, Raleigh, 1987

B.S. Chemical Engineering, Jadavpur University, Calcutta, India, 1985

Positions Held97-pre **Professor**, Materials Science and Engr., University of Florida94-pre **Director**, Characterization Research Instrumentation and Testbed (CRIT) Facility,
Engineering Research Center (ERC), University of Florida96-pre **Thrust Leader**, Chemical Mechanical Planarization (CMP), ERC Univ of Florida94-pre **Thrust Leader**, Engineered Particulates, ERC, Univ. Florida95-97 **Associate Professor**, Materials Science and Engr., University of Florida90- 94 **Assistant Professor**, University of Florida, Gainesville, FL**Awards/Honors**2000 - **Distinguished Visiting Scientist**, NIRIM, Tsukuba, Japan1999 **Distinguished Visiting Professor**, National University of Singapore, Singapore1998 **Distinguished Visiting Professor**, University of Osaka, Osaka, Japan1995 **Hardy Gold Medal** from TMS/AIME for Outstanding Contributions in Materials Science1994 **NSF Young Investigator Award**94-97 **Visiting Fellow**, Center for Ultrafast Optical Science (CUOS), University of Michigan1993 **IEEE Senior Member Award**1991 **IBM Faculty Development Award**1989 **MRS Best Graduate Student Award**1985 **Alumni Gold Medal** for Best Overall Graduating Senior from the University1985 **Laha Silver Medal** for Best Graduate from College of Engineering**Publications:**

Over 293 papers (> 268 published/in print & 25 submitted for various materials science and engineering journals (*Science*, *Physical Review B*, *Applied Physics Letters*, *Journal of Materials Research*, *Materials Science and Engineering B*, etc.) and Conference Proceedings. Published over 32

original, *principal author papers in App. Phys. Lett.* (The most cited electronic materials/applied physics based journal), and 7 papers published in *Physical Review B*

Invited and Contributed Talks

Presented more than 110 invited talks at international conferences (MRS, SPIE, TMS, APS, ASME, etc.) and academic and research institutions (MIT, Columbia, Purdue, ORNL, Westinghouse, etc.). Also group presented over 250 technical papers at international conferences

Books and Guest Editorships (Edited 5 books & Guest Editor of 5 Journal Issues)

- (1) R. K. Singh, D. Norton, J Cheung and J. Narayan and L.D. Laude , *Eds "Laser Processing of Materials: Fundamentals and Advanced Applications*, MRS Proceedings Vol 397, Pittsburgh, PA, 1996
- (2). N. M. Ravindra and R.K. Singh, "*Transient Thermal Processing of Materials*", TMS, Warrendale April. 1996
- (3). K. Gonsalves, M. Baraton, J.. Chen, J. Akkara, R. K. Singh and H. Hofmann , "*Surface Controlled Nanoscale and Microscale Materials for High Value Added Applications*", MRS Proceedings Vol 501 , Pittsburgh, PA, March 1998
- (4). R.K. Singh, D. Lowdnes, J. Narayan, D. Chrisey , T. Kawai, and E. Fogarassy, Editors, *Advances in Laser Ablation of Materials*", MRS Proceedings for Spring 1998 .
- (5). R. K. Singh and D. Kumar, "*Advances in Pulsed Laser Deposition of Thin Films*", Kluwer publishers, (1998)
- (1) *Guest Editor* of September 1994, Vol 23 issue of *Journal of Electronic Materials* titled " *Novel Issues in Photonic Materials*"
- (2) *Guest Editor* of Jan,96, Vol 1 issue of *Journal of Electronic Materials* titled "*Ion and Laser Beam Processing of Electronic Materials*"
- (3) *Guest Editor* of Materials Science and Engr. B, on *Laser Processing of Electronic Materials*, Jan 1997
- (4) *Guest Editor* of November 1997 Issue of *Journal of Electronic Materials* on "*Low Energy Beam Processing of materials.*"
- (5) *Guest Editor* of September 1998 Issue of *Journal of Electronic Materials* on "*Chemical-Mechanical Polishing of Semiconductors.*"

Teaching Accomplishments

Developed four new courses: "Beam-Solid Interactions", "Thin Films" & "Math. Methods", "Survey of Materials Analysis" in the graduate MS&E program
 Graduated 12 Master's and 10 Ph.D Students; Presently thesis advisor to 9 Ph. D Students
 8 students awarded best paper/fellowships for their undergraduate/graduate research projects.
 Developing CD-ROM materials and multi-media classroom for the NSF ERC project.
 Established ParTiN (Particle technology) Hypertext Network for educational & ERC programs on the WWW (World Wide Web)

Corporate Interactions

Direct Research Interactions with several companies including IBM, Intel, Motorola, Ashland Chemical, Westinghouse, Lucent Technology, Applied Materials, Sony, Glaxo Wellcome, Lockheed Martin, Astra Zeneca, Purdue Pharma
 Licensing Discussions with Sony, Nara Machinery, Astra Zeneca, Glaxo, etc.
 Corporate funding over 200 K/yr .

Copyrighted Softwares (3 copyrighted softwares) including

(A) **SLIM** (*Simulation of Laser Interaction with Materials*, 36,000 coded lines, 1992) software.

This first of its kind software is being used by more than 50 R&D groups (IBM, LANL, ORNL, etc.) in the world. This software calculates the transient thermal induced laser effects like melting, crystallization and ablation of materials. This software has had sales greater than \$ 60 K worldwide in the last four years. Two new versions (one based on DOS C++ and the other on Windows platform) have been developed..

Patents (from a total of 30 disclosures:[14 patents, 10 awarded/pending(final stage) and 4 filed])

(1) ***High Surface Area Metals and Ceramics*** [US Patent 5,473,138] . A unique laser technique has been developed to increase the surface areas of ceramics, metals and composites. This technique involves the use of multiple-pulse laser irradiation under controlled energy window conditions.

(2) ***Enhanced Chemical Vapor Deposition of Diamond*** [US Patent 5,485,804 {1996}, Filed for worldwide patent} Novel colloidal method for large area nucleation, of diamond. *This method has been used to make the world's largest single monolithic piece of diamond which has a diameter greater than 11" and weighs over 1600 carats.*

Conference Chairs [Organized 16 international conferences on innovative processing and characterization of materials]

(1) Chair of Symposium. on "Beam Processing of Materials", ***TMS/AIME Winter meeting***, Chicago Nov 92;

(2) Co-Chairman of Symposium on "Innovative Processing of Electronic and Photonic Materials" ***TMS/AIME Annual Meeting***, Denver, Feb 1993;

(3) Chairman of Conference on "Advanced Laser Processing of Materials" ***Engineering Foundation*** Conf., Palm Coast, FL, May 1-6 1994:

(4) Chair of Symposium on " Ion Beam Processing of Materials" ***TMS Spring Meeting***, Las Vegas, Feb 1995

(5) Co-Chair of symposium on "Laser Processing of Materials" ***American Physical Society***, San Diego, March 1995

(6) Chair of Symposium on " Advanced Laser Processing of Materials: Fundamentals and Advanced Applications" ***MRS Meeting***, Boston Nov 1995

(7) Co-Chair, Symposia on "Transient Thermal Processing of Materials", ***TMS Annual Meeting***, Anaheim, CA Feb, 1996

(8) Chairman of symposium on, " Low Energy Beam Processes", ***TMS Annual Meeting***, Orlando , FL Feb, 1997

(9) Chairman of symposium on " Particulate Coatings", ***MRS Fall Meeting***, Boston November, 1997

(10) Co-Chairman of " Laser and Ion Beam Processing of Materials", ***International Union of Materials Research Societies (IUMRS)***, Chiba, Japan, September 1997

(11) Co-Chairman, " Transient Thermal Processing of Materials , ***TMS Annual Meeting***, San Antonio, Feb 1998

(12) Co-Chairman, " Chemical Mechanical Planarization of Materials Symposia, ***TMS Annual Meeting***, San Antonio, Feb 1998

(13) Chairman, " Advances in Pulsed Laser Ablation of Materials", ***MRS Spring Meeting***, San Francisco, April, 1998

- (14) Co-Chairman, "Particulate Coatings" **5th World Congress on Particle Science and Technology**, Brighton UK, July 1998
- (15) Co-Chairman "Rapid Thermal Processing of Materials"- **European MRS Meeting**, Strasbourg, June, 1998
- (16) Chairman, "Chemical Mechanical Polishing Symposia", **MRS Spring Meeting**, San Francisco, April 2000

Invited Review Articles

- 1. "Pulsed Laser Deposition of Thin Films", *Materials Science and Reports* in March, (1998)
- 2. "SLIM,. A Personal Computer Based Simulation of Laser Interaction With Materials", *J Journal of Materials*, **44**, 20 (1992)
- 3. " Pulsed Laser Deposition and Processing of Superconducting Thin Films", *J. of Materials* **43**, 13 (1991)

Book Chapters

- 1. D. Gilbert and R. K. Singh, " Boron Nitride Interfaces", in " *Properties of Group III Nitrides*, Edited by James Edgar, *Imspec* publication, London 1995
- 2. R. K. Singh, " Raman Based Optical Properties of YBaCuO Surfaces", in " *Optical Properties of Materials*", Eds R. Hummel, CRC Press, 1996
- 3. R.K. Singh and D. Kumar, "Pulsed Laser Deposition of Superconducting Thin Films", *Materials Science and Engr. Reports* (in press, 1996)
- 4. R. K. Singh and D. Kumar, " Thermal Annealing of Semiconductors", *Encyclopedia of Applied Physics*, VCH Publishers (1996)
- 5. D. Gilbert and R. K. Singh, " Diamond Deposition for Electronic Applications", Eds . S. Pearton on *Wide Band Gap Semiconductors*", VCH Publishers 1997

Reviews

Reviewer for NSF, DOE, Physical Review B, Materials Science and Engineering, Journal of Applied Physics, Applied Physics Letter, Journal of Materials Research and Physica C.
Invited to serve as panel members for several NSF initiatives

Memberships and Committee Chairmanships:

Member of MRS, ASM, TMS, IEEE (senior member), APS, AICHE, SPIE
Chairman: Thin Films and Interfaces Committee , TMS (1993 -1996)
Member: Laser Processing of Materials Committee, CLEO

Institutional Impact

- (A) Helped establish the 60 million, NSF Funded **Engineering Research Center (ERC)** on particle science and technology at the University of Florida:
- (B) Leader of team for the NSF MRSEC Proposal submitted in 1995 & 1997
- (C) Established cross-disciplinary multi P.I programs in Rapid Thermal Processing, Particle Coating Technology, Radiation Damage in Electronic Devices, and Beam Processing of Materials:
- (D) Established industrial and scientific collaborations with various institutions:
- (E) Established international collaborations with

(i) CNRS, Strasbourg France, (ii) University of Melbourne, Australia, (iii) University of Osaka, Japan, (iv) Keio University, Japan, (v) EPFL, Lausanne, Switzerland, (vi) Unicamp, Sao Paulo Brazil, (vii) National University, Singapore

Appendix E
DECLARATION UNDER 37 C.F.R. §1.132
OF
PROFESSOR TERRY BRICKER

Resume of Dr. Bricker is also attached.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Kambe et al.

Serial No.: 08/962,362

Filed : October 31, 1997

For : PHOSPHORS

Docket No.: N19.12-0006

Group Art Unit: 2879

Examiner: M. Day

DECLARATION UNDER 37 C.F.R. § 1.132

Express Mail: EL63605030505Date of Deposit: October 10, 2000

BOX AF
Assistant Commissioner for Patents
Washington, D.C. 20231

I, Terry M. Bricker, Ph.D., hereby declare as follows:

1. I am presently the Mooreland Family Professor of Basic Sciences, Department of Biological Sciences and an adjunct Professor of Chemistry at Louisiana State University, Baton Rouge, Louisiana.
2. I received my Ph.D. degree in 1981 in Botany from Miami University.
3. I have been on the faculty at the Louisiana University since 1987. I was promoted to Associate Professor with tenure in 1990 and to full Professor in 1994. A copy of my Resume is attached.
4. I have been Visiting Professor at the University of Illinois and Michigan State University. I am the author or co-author of many scientific articles, conference proceedings and review papers. I have served on the editorial board of Plant Physiology and the Annual Reviews of Plant Physiology and Plant Molecular Biology.
5. I have no financial interest in NanoGram Corporation or in the present patent application.

6. I have extensive experience in separation technology applied to biological nanoparticles, in particular, proteins and DNA. I and coworkers in my laboratory continuously use several forms of chromatography in the separation and purification of proteins. I have used chromatography and protein purification techniques throughout my career.

7. I have read carefully U.S. Patent 5,442,254 to Jaskie (the Jaskie patent). I have evaluated the description of particle separation in the Jaskie patent based on my extensive expertise in separation technology generally.

8. The isolation method of the quantum particles describe in the Jaskie patent at column 7, lines 28-40 relies on the use of capillary action to separate particles of different diameters. Specifically, particles with a diameter range of about 10 to 100 angstroms are suspended in a liquid, and the liquid is allowed to move up a cloth by capillary action. The authors argue that the distance which various particles migrate up the cloth is directly proportional to their size. The authors further suggest that at any given height up the cloth all of the particles will be the same size. Thus, the authors are describing a chromatographic system which they allege will differentially fractionate the particles based on size.

9. The separation techniques described in the Jaskie patent will not separate different size classes of quantum particles. First, a mixture of different sized particles is continuously loaded onto the cloth. Even assuming for argument that the different sized particles migrate at different speeds, additional particles are continuously loaded behind the migrating edge of initially loaded particles. Thus, the particles are continuously remixed with particles of other sizes as additional particles are loaded onto the cloth. This remixing occurs for every size class of particles. Significantly, the technique will not work because no cloth is known with the necessary properties to differentially interact with different sized particles.

Traditional thin layer chromatography is based on differential solubility constants for the chemicals being separated. Due to different solubility constants, solutes migrate at different rates as the solvent is taken up by capillary action.

10. Any chromatographic separation approach relies on 1) the properties of the liquid that the particles are suspended in, 2) the surface characteristics of the cloth, 3) the surface properties of the quantum particles, and 4) the size of the quantum particles. The patentees provide no direct information on the first three of these categories. Based on the discussion in the previous paragraph in column 7 of the patent, perhaps one can assume that the solvent is water. However, the surface properties of the cloth used in the described separation is critical. Separation in any chromatographic system is dependent on differential partitioning of the solutes, i.e., the quantum particles, between a mobile phase, the water, and a stationary phase, the cloth. However, no such cloth exists. For separation of biological macromolecules, such separation by size is the purview of gel filtration chromatography. Even in gel filtration fractionation systems, the relatively small differential partitioning coefficients observed prevent true high resolution separations. In this particular instance, one must necessarily obtain high degrees of dimensional resolution of the quantum particles to achieve wavelength selectivity. The required dimensional resolution cannot be achieved by the methods presented in column 7, lines 28-40 of the Jaskie patent.

11. The addition of an electric field would not overcome these problems. First, the authors do not make any claim that the surface charge on the particles is directly proportional to the size. The authors do not describe the means of attaching the electric current or even if the current is applied axially or perpendicular to the capillary flow. Critically, the authors do not describe the properties of the cloth even though the properties of the cloth would critically effect the separation in

the presence of the electric field. While gel electrophoresis is used to separate biological macromolecules using electric fields, these separation are performed in polymer gels, not cloth, that have been developed for the specific purpose of separating biological macromolecules. The gel acts as a sieve allowing the fractionation of the biological macromolecules by size, surface charge and steric properties. No cloth exists with the required sieving properties. Even the protocols effective to separate different types of biological macromolecules, such as water soluble proteins, membrane proteins and nucleic acid fragments, are significantly different from each other. Inorganic particles are very different with respect to chemical properties and chemical structure from biological macromolecules. The Jaskie patent provides no information that guides anyone trying to adapt these biochemical methods to the separation of quantum particles.

Thus, a person familiar with the separation technologies could not separate Jaskie's quantum particles based on information provided in the Jaskie patent.

12. I declare that all statements made herein that are of my own knowledge are true and that all statements that are made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date:

10/8/00

By:



Terry M. Bricker, Ph.D.

RESUME

Dr. Terry M. Bricker

Position: Professor

EDUCATION:

INSTITUTION AND LOCATION	DEGREE	YEAR CONFERRED	FIELD OF STUDY
Miami University Univ. Missouri	Ph.D. Postdoctoral Work	1981 1981-1985	Botany Biological Sciences

RESEARCH AND PROFESSIONAL EXPERIENCE:

National Science Foundation, IGERT Pre-proposal Panel, 9/00.
Interim Chairperson, Department of Biological Sciences, 6/99-8/00.
Moreland Family Professor of Basic Sciences, Department of Biological Sciences,
4/99-Present
Invited Speaker, Gordon Research Conference Photosynthesis-Biochemical
Aspects, 6/99.
Editorial Committee, Annual Reviews of Plant Physiology and Plant Molecular
Biology - Vol. 52, 10/98.
Adjunct Professor, Department of Chemistry, Louisiana State University, 3/98-
Present.
Professor, Department of Biological Sciences, Louisiana State University, 7/97-
Present.
Professor and Chairman, Department of Microbiology, Louisiana State
University, 7/95-6/97.
National Science Foundation, Research Training Grant site visit team to Penn
State University, 6/96
National Science Foundation, Research Training Grant Advisory Panel, 4/96
Discussion Leader, Gordon Research Conference, "Photosynthesis - Biochemical
Aspects", 8/96
Visiting Professor, Department of Plant Biology, University of Illinois 1/95-6/95.
Professor, Department of Plant Biology, Louisiana State University, 8/94-7/95.
Visiting Faculty, Plant Biochemistry Intensive Summer Course, MSU-DOE Plant
Research Laboratory, Michigan State University, 6/94.
Associate Professor, Department of Botany, Louisiana State University, 8/90-7/94.
Invited Speaker, Gordon Research Conference, "Photosynthesis - Biochemical
Aspects", 8/93.
Director, Protein Analysis Center, College of Basic Sciences, 7/90-8/92.

Dr. Terry M. Bricker
Page Two

Graduate Coordinator, Department of Botany, Louisiana State University, 9/89-8/92.

Department of Energy, *Ad hoc* Advisory Panel Basic Energy Biosciences, 1991.

National Science Foundation Molecular Biochemistry Program Advisory Panel, 10/90-4/94.

Assistant Professor, Department of Botany, Louisiana State University, 8/87-8/90.

Editorial Board, *Plant Physiology*, 1/90-7/92.

Assistant Professor, Department of Chemistry, University of Southern Mississippi, 1/85-8/87.

Publications (last five years, only)

56. Rosenberg, C., Christian, J., Bricker, T. M. and C. Putnam-Evans, "Site-Directed Mutagenesis of Glutamate Residues in the Large Extrinsic Loop of the Photosystem II Protein CP 43 Affects PS II Assembly." To appear *Biochemistry*.
55. Bricker, T.M. and Frankel, L.K. "The Role of Carboxylic Acid Residues on the Manganese-Stabilizing Protein in its Binding to Photosystem II." To appear *Biochemistry*.
54. Wu, J., Masri, N., Lee, W., Frankel, L.K. and T.M. Bricker, "Directed Random Mutagenesis in the Large Extrinsic Loop of the CP 47 Protein of Photosystem II." *Plant Molecular Biology* 39,381-386 (1999).
53. Knoepfle, N., Bricker, T.M., and Putnam-Evans, C., "Site-Directed Mutagenesis of the Basic Residues ³⁰⁵R and ³⁴²R in the CP 43 Protein of Photosystem II Affects Oxygen-Evolving Activity in *Synechocystis* 6803." *Biochemistry* 38,1582-1588 (1999).
52. Bricker, T.M., Morvant, J., Masri, N., Sutton, H. and Frankel, L.K., "Isolation of an Oxygen-Evolving Photosystem II Preparation from *Synechocystis* 6803 using a Histidine-Tagged Mutant of CP 47." *Biochimica et Biophysica Acta* 1409, 50-57 (1998).
51. Zubrzycki, I.Z., Frankel, L.K., Russo, P.S. and T.M. Bricker, "Hydrodynamic Studies on the Extrinsic "33 kDa" Protein of Photosystem II." *Biochemistry* 37,13553-13558 (1998).
50. Ghanotakis, D., Tsiotisz, S. and T.M. Bricker, "Polypeptides of Photosystem II: Structure and Function." In: *Plant Photobiology: Photosynthesis and Photomorphogenesis* (Singhal, G.S., Renger, G., Sopory S.K. Irrgang, K.-D. and Govindjee, eds.) pp. 264-291, Narosa Publishing House, New Delhi, India (1998).
49. Bricker, T.M. and L.K. Frankel, "Structure and Function of the 33 kDa Extrinsic Protein of Photosystem II." *Photosynthesis Research* 56:157-173 (1998).

48. Bricker, T.M., Putnam-Evans, C. and J. Wu, "Mutagenesis in the Study of the Structure and Function of Photosystem II." *Methods of Enzymology* 297:320-337 (1998).
47. Qian, M., Al-Khaldi, S., Putnam-Evans, C., Bricker, T.M. and R.L. Burnap, "Photoassembly of the Photosystem II (Mn)₄ Cluster in Site-Directed Mutants Impaired in the Binding of the Manganese-Stabilizing Protein." *Biochemistry* 36,15244-15252 (1997).
46. Putnam-Evans, C. and T.M. Bricker, "Site-Directed Mutagenesis of the Basic Residue ³²¹R to ³²¹G in the CP 47 Protein of Photosystem II Alters the Chloride Requirement for Growth and Oxygen-Evolving Activity in *Synechocystis* 6803." *Plant Molecular Biology* 34:455-463 (1997).
45. Putnam-Evans, C., Wu, J., and T.M. Bricker, "Site-Directed Mutagenesis of the CP 47 Protein of Photosystem II: Alteration of Conserved Charged Residues within Lethal Deletions in the Large Extrinsic Loop of CP 47", *Plant Molecular Biology* 32:1191-1195 (1996).
44. Wu, J., Putnam-Evans, C., and T.M. Bricker, "Site-Directed Mutagenesis of the CP 47 Protein of Photosystem II: ¹⁶⁷W in the Lumenally Exposed Loop C is Required for Photosystem II Assembly and Stability.", *Plant Molecular Biology* 32,537-542 (1996).
43. Bricker, T.M. and Demetrios Ghanotakis "The Structure and Function of the Oxygen-Evolving Complex" In: *Advances in Photosynthesis, Vol. 4, Oxygenic Photosynthesis: The Light Reactions*, pp. 113-136, Yocum, C.F. and Ort, D.R., eds. (1996).
42. Putnam-Evans, C., Wu, J., Burnap, R. Whitmarsh, J. and T.M. Bricker, "Site-Directed Mutagenesis of the CP 47 Protein of Photosystem II Alteration of Conserved Charged Residues in the Domain ³⁶⁴E-⁴⁴⁴R", *Biochemistry* 35,4046-4053 (1996).
41. Leuschner, C. and T.M. Bricker, "Interaction of the 33 kDa Extrinsic Protein with Photosystem II: Rebinding of the 33 kDa Extrinsic Protein to Photosystem II Membranes which Contain Four, Two, or Zero 40. Manganese per Photosystem II Reaction Center." *Biochemistry* 35,4551-4557 (1996).
40. Frankel, L.K. and T. M. Bricker, "Identification of Domains on the 33 kDa Extrinsic Protein which are Shielded from NHS-Biotinylation by Intrinsic Photosystem II Components", *Biochemistry* 34,7492-7497 (1995).

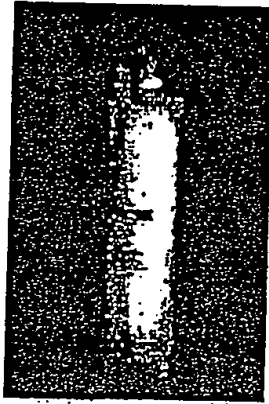
APPENDIX F
PRODUCT LITERATURE FROM MILLIPORE CORPORATION



MILLIPORE

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Planargard Disposable Filters



Planargard Disposable filters offer convenience and versatility when filtering CMP slurries, with full compatibility for both oxide and metal slurries. The unique design, employing standard Flaretek fittings and a fully integrated polypropylene filter and shell, provides automatic draining capabilities, reduced holdup volume, and minimized exposure during filter change-out. Planargard Disposable filters avoid problems caused by non-standard fittings, and allow users to choose a range of options from maximum filter life to fewer wafer defects. Planargard Disposable filters — the result of Millipore's extensive applications expertise — represent the latest advances in proven filtration capabilities.

Specifications

Materials of Construction

All polypropylene construction with no O-rings or elastomeric seals.

Connections

Inlet/Outlet

1/2 in Flaretek fittings, 1 in Flaretek fittings

Vent/Drain

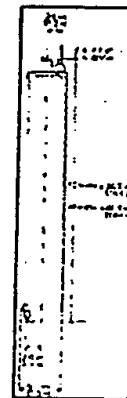
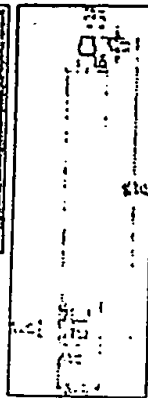
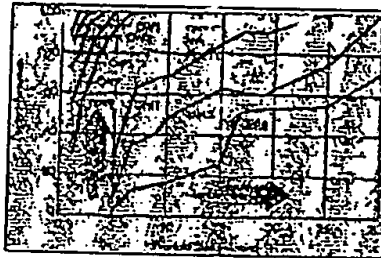
1/4 in Flaretek fittings

Operating Conditions

Maximum Pressure

4.1 bar (60 psi) at 25 °C

ORDERING INFORMATION									
C	M						0	6	
		Filter Choice		Length		Package Quantity			
		P3 P5 P7 P9 11 13 16		1 = 10 in 2 = 20 in		06 = 6 per package			
		I = In-Line U = U-Line		1 = 1 in Flaretek inlet/outlet fittings (In-Line only) 5 = 1/2 in Flaretek inlet/outlet fittings (U-Line only)					
Note: Filter choices CM13 and CM16 are not available in the U-line configuration.									



Planargard disposable In-Line filter with 1 in Flaretek inlet/outlet fittings and 1/4 in Flaretek vent/drain fittings

Planargard disposable U-Line filter with 1/2 in Flaretek inlet/outlet fittings and 1/4 in Flaretek drain fittings

[For a complete technical data sheet, click here](#)

How to place an Order

For application questions, contact Millipore Technical Service

For more information, contact your local Millipore Application Specialist

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Planargard

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Millipore Planargard Filters



Description

- Graded-density filter removes agglomerated large particles before they can scratch wafer surfaces
- Removes disruptive particles without removing small working particles, ensuring consistent slurry delivery to your process
- All polypropylene construction provides excellent compatibility with high and low pH slurries

Applications

- Point-of-use and bulk filtration of ILD slurries for chemical mechanical planarization (CMP)

Specifications

Materials

All-polypropylene construction
O-rings
EPR

Cartridge Dimensions

Diameter
70 mm (2.75 in)
Length
10": 264 mm (10.4 in)
20": 512 mm (20.2 in)
30": 761 mm (30 in)

Connections

Code 0 2-222 double O-ring

Operating Conditions

Maximum Forward Differential Pressure

4.8 bar (70 psi) at 20 °C

Filter Selection Guide

Filtration Objective	Location	Suggested Product
Minimize wafer defects	Point of use	CMP1, CMP3, CMP5
Extend filter life	Point of use	CMP7, CMP9, CM11
Remove gross contaminants	Point of use	CM13
Reduce large particles formed in shipping container	Intake	CM14, CM16, CM18
Reduce large particles and gels formed during dilution	Post-dilution	CMP9, CM11, CM13
Continuous cleaning	Recirculation loop	CM14, CM16, CM18

ORDERING INFORMATION

C M O E O 6

Filter Choice
P1
P3
P5
P7
P9
11
13
14
16
18

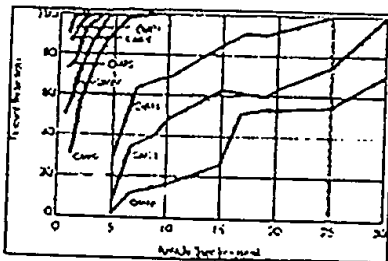
Length
1 = 10"
2 = 20"
3 = 30"

Package Quantity
06 = 6 per package

Cartridge Code
0 = Code 0
(2-222) O-Ring

O-Ring Material
Delivered with EPR
O-Rings installed
on cartridge

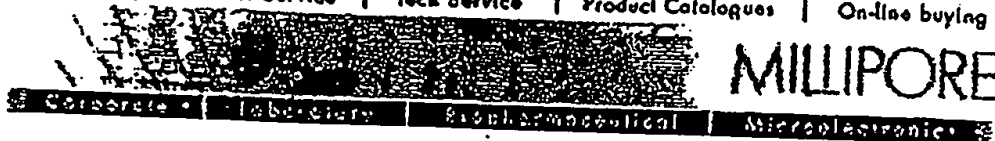
Note: All Planargard filters are delivered with EPR,
Code 0 (2-222) O-Rings installed.
All Planargard filters are shipped 6 per package.



Actual Particle Retention in Oxide SlurryXXX

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Guide to Millipore Products for CMP Processes

The CMP slurry filtration challenge is to remove large particles and agglomerates from slurry that can cause defects, without changing slurry performance.

Point-of-Use Filtration



CMP Slurries introduce millions of abrasive particles to the wafer, potentially causing scratches on the surface. Point-of-use filtration is the most effective location for reducing the number of defect-causing particles. We have seen up to a ten-fold reduction in light point defects by using Planargard filters at the point-of-use.

For more applications information, see our Millipore technical document, MA071 POU Filtration of Silica-Based CMP Slurries Using Planargard Filters

by Zhenwu Lin, Joseph Zahka, Geanne Vasilopoulos.

Point of Use Filtration Selection Guide

Find the type of slurry used in your process and select the filter for specific product information.

Slurry Type

Recommended Filter

Fumed Silica Oxide
(Diluted Cabot[®] SS25 and SC1,
SS12, SC112; Rodel[®] ILD1300,
ILD1200)

- Planargard Cartridge Filter,
membrane type CMP5
- Planargard Disposable
Filter, membrane type
CMP5
- Planargard Cartridge Filter,
membrane type CMP3
- Planargard Disposable
Filter, membrane type
CMP3

Colloidal Silica Oxide
(Klebosol[®] slurries)

- Planargard Cartridge Filter,
membrane type CMP5
- Planargard Disposable
Filter, membrane type
CMP5
- Planargard Cartridge Filter,
membrane type CMP3
- Planargard Disposable
Filter, membrane type
CMP3

Silica-based Metal
(Cabot SSW200)

- Planargard Cartridge Filter,
membrane type CMP5
- Planargard Disposable
Filter, membrane type
CMP5

Metal (non-silica based)
<2% solids
>2% solids

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Liquid Application Notes Database

Millipore MicroElectronics Division Technical Document MA071

POU Filtration of Silica-Based CMP Slurries Using Planargard Filters

Zhenwu Lin, Joseph Zahka, Geanne Vasilopoulos

Introduction

Chemical Mechanical Polishing (CMP) has become an enabling technology in semiconductor device manufacturing. The CMP process uses submicron (30 - 200 nm) silica slurries at a typical concentration of 10-30% solids. Typical silica slurries can contain a small number (104 to 106 counts/ml) of > 1.0 micron particles, which could potentially cause defects (microscratches) on the planarized wafer surfaces. The slurry solution presents unique challenges in delivery, filtration, and particle measurement.

The silica slurries used in the CMP process are stabilized suspensions of fine particles, typically 30 to 200 nm in size with concentrations ranging from 10 to 30%, in aqueous solutions with a specific pH. These slurries, which may need on-site dilution in the FABs, are applied directly onto the rotating pad to polish wafers and achieve global planarity. Fumed silica-based and "colloidal" silica-based slurries are the two families that are commercially available.

Fumed silica slurry is manufactured in two steps: 1) production of fumed silica (three-dimensional branched chain aggregates) by the vapor phase hydrolysis of silicon tetrachloride in a hydrogen oxygen flame [2]; 2) dispersion of fumed silica in aqueous medium with certain additives. Commercial fumed silica CMP slurries normally contain silica aggregates with mean particle sizes ranging from 100 nm to 200 nm. Figure 1a shows a SEM picture of typical silica aggregates.

"Colloidal" silica is produced from a dilute aqueous solution of water glass through deionization/nucleation, polymerization, particle growth and concentration steps [3]. All process steps are in the liquid phase. The silica particles formed are normally spherical. Commercial colloidal silica CMP slurries have particles with mean sizes ranging from 30 nm to 50 nm. Figure 1b shows a SEM picture of typical colloidal silica particles.

Filtration Needs

The typical specification for commercial silica slurries includes percent solids, pH, specific gravity, mean particle size and general (bulk) particle size distribution. However, a small number of "large" particles (>1µm) have been found which fall outside of the specified size distribution. These particles, which can be aggregates, agglomerates. SEM images shown in Figures 2A and 2B confirm their existence. These large particles may come from agglomeration or local drying of slurry on shipping containers and in the distribution system. Gels may form due to pH shocks during dilution or

temperature fluctuations during shipment and storage.

There is no definitive information available on what size or type of particles can cause microscratches and particle contamination on wafer surfaces. However, higher numbers of "large particles" have been found to cause higher incidence of microscratches and particle contamination on polished wafers. Slurry filtration has proven to be beneficial in reducing wafer defects and increasing yields in CMP processes[1].

A control oxide CMP polishing experiment was conducted using a commercial fumed silica slurry contaminated with 5 m silica particles (~ 104 particles/ml). The polishing were performed on an IPEC/Westech 472 tool using a standard oxide CMP recipe. The bare wafers were deposited with 1000 nm PECVD SiO₂ before polishing. Figure 3 and 4 show the surface scan results on the wafers polished with and without point-of-use filtration. A ten-fold reduction of light point defects was achieved by using a Planargard CMP5 filter at the POU.

Slurry Characterization

The main challenge of slurry filtration is to selectively retain the small number of defect-causing "large" particles (i.e., 104 to 106 counts/ml greater than 1 μ m) without retaining the desirable, small particles (30 to 200 nm) present in very high concentration (> 1015 counts/ml). There should be no measurable changes to the slurry's percent solids concentration and bulk particle size distribution before and after filtration. Therefore, filters to be used in CMP slurry filtration should be evaluated *in slurry* to validate their performance for the following attributes:

- Retention efficiency for "large" particles
- % solids content and bulk particle size distribution before and after filtration
- Throughput (Lifetime)

1. Detection of "Large" Particles

Quantitative determination of "large particles" is required to determine filter retention and a correlation between large particle concentration and wafer defects. There is no commercial particle counters available that can be used to detect the large particles (104 to 106 counts/ml >1 μ m) in the presence of bulk slurry particles (> 1015 counts/ml), without substantial sample dilution. Various particle counters were evaluated to determine their ability to detect the large particles with maximum tolerance to high concentrations of small particles (minimum sample dilution) and with ease of operation. A light scattering instrument was selected for the slurry application.

A typical schematic of the particle counting system, shown in Figure 5, includes continuous on-line dilution of slurry. The dilution factor should be high enough to minimize the interference caused by small particles in the slurry. Figure 6 shows a typical profile of large particle concentrations in oxide slurry before and after filtration. Filter retention for particles of a specific size can then be calculated based on the particle concentration before and after filtration.

2. Measurement of Bulk Particle Size Distribution and Percent Solids

Bulk particle size distribution (PSD) can be measured by many techniques [4]. The two most commonly used techniques in CMP slurries are light scattering and chromatography (i.e., capillary hydrodynamic fractionation, or CHDF). The light scattering instrument used for slurry PSD measurement is based on photon correlation spectroscopy (PCS), also referred to as quasi-elastic light scattering (QELS) or time-dependent light-scattering. With PCS, the size information is obtained from the time dependent fluctuation of scattered intensity due to concentration fluctuations resulting from Brownian motion of particles[4].

CHDF is based on the size exclusion effects that occur when a dispersion of particles flows through a

capillary tube. Laminar flow in the capillary tube has a parabolic velocity profile. Smaller particles can reach the slower streamline close to the tube wall due to Brownian motion, while larger particles cannot. Therefore, large particles exit the capillary tube faster than smaller ones. CHDF can be used to measure particles between 15 nm and 1 micron with up to 1% solids [5]. This method was used for slurry particle size distribution measurement.

Another PSD instrument is based on acoustic attenuation spectroscopy. When acoustic waves propagate through a medium with suspended particles, the acoustic beam will be further attenuated by the particles by a variety of mechanisms. The acoustic attenuation spectrum can be detected and then inverted to obtain a mean particle size, a particle size distribution, and a dispersion concentration.

The percent solids in slurry can be calculated by drying a slurry sample of known weight.

Performance of Planargard TM Filters

Filter performance should be evaluated in slurry to determine: (a) retention efficiency for large particles; (b) percent solids content and bulk particle size distribution before and after filtration; (c) filtration throughput. This information is necessary for CMP process engineers to implement proper filtration.

Conventional microporous membrane filters will not work due to the high solids concentration in the slurry that forms a cake and plugs membrane quickly. Graded-density non-woven depth filters are preferred for this application. Filtration experiments were conducted with all-polypropylene graded-density PlanargardTM filters using silica slurry in a single-pass configuration to simulate point-of-use applications. Feed and filtrate samples were taken and analyzed for large particle concentration, percent solids, and bulk particle size distribution. The filter retention efficiency is defined as:

Concentration in feed - Concentration in filtrate

$$\text{Retention} = \frac{\text{Concentration in feed} - \text{Concentration in filtrate}}{\text{Concentration in feed}} \times 100\%$$

The retention efficiency curves for various Planargard filters are shown in Figure 7.

Throughput was measured based on the differential pressure across the filter as a function of filtered volume. A typical plugging curve is illustrated in Figure 8, which shows a gradual increase in differential pressure across the filter. The differential pressure increases slowly initially, but then climbs rapidly as the filter reaches the end of life. Data analysis proves that the plugging process follows the complete plugging mechanism, which can be represented by a linear relationship between the inverse differential pressure across the filter (or DP_{min}/DP in dimensionless form) and filtration volume. In this mechanism, the pressure drop across the filter increases slowly at the beginning, which will then increase exponentially. The importance of understanding the plugging process is to determine the filter change out time before the pressure drop reaches the region of exponential increase.

To maintain process control the filter should not affect the slurry's composition and the filter should have consistent retention throughout its useful lifetime. Figure 9 shows that filter retention remains fairly constant throughout its lifetime. Table I shows that the filter does not change the percent solids and mean particle size in the slurry, which is extremely important for a CMP process. As long as the solids concentration is not changed before and after filtration, the filter will not alter the bulk particle size distribution. Figure 10 shows the slurry's bulk particle size distribution in the feed and filtrates at 60% and 95% of the CMP5 filter's throughput.

Implementation Strategy

Implementation of silica slurry filtration depends on CMP process requirements, space availability, and the characteristics of the slurries. A number of field tests have demonstrated that point-of-use (POU) filtration at the tool can provide the most benefit in wafer defect reduction. Table II lists the normalized test results for oxide CMP with POU filtration.

Other filtration locations are at post-dilution, in the distribution loop, and at the slurry supply drum/tote.

It is strongly recommended that the implementation should start at POU filtration with higher retention filters to realize the maximum defect reduction benefits during CMP process development and qualification. To optimize the process, filtration at other locations can be used to supplement POU filter(s) and potentially to extend the life of POU filters.

Conclusion

"Large particles" have been detected in CMP slurries using an optical particle counter. SEM evaluation of slurry particles on membrane filters confirmed their existence. These defect-causing large particles may come from agglomeration, local drying of slurry on shipping containers and in the distribution system, and gel formation due to pH shocks during dilution and temperature fluctuations.

Graded-density depth filter can be used effectively to remove the defect-causing large particles without measurable change to slurry composition. Field test data have demonstrated the benefits of filtration on wafer defect reduction during CMP processes.

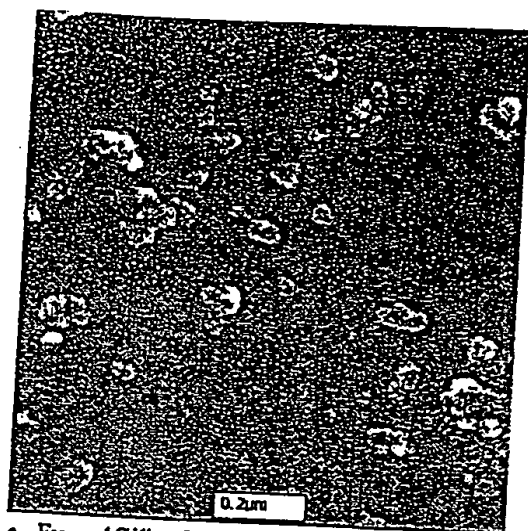
The optimal strategy to implement filtration in CMP processes can be dependent on site, process, and slurry type. POU filtration with higher retention filters is recommended to realize maximum defect reduction benefits during CMP process development and qualification.

References

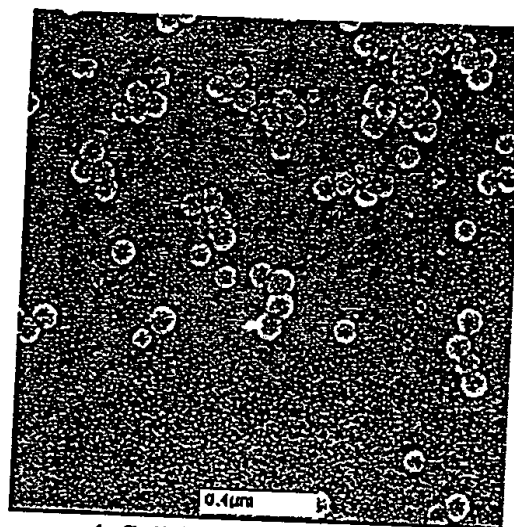
1. Nagahara, R., et al, The effect of slurry particle size on defect levels for a BPSG CMP process, Proceedings of the CMP Users Group, Vol. 1, No.1, July, 1996
2. CAB-O-SIL Untreated Fumed Silica Properties and Function, Technical brochure, Cabot Corp
3. Yoshida, A., Silica Nucleation, Polymerization, and Growth Preparation of Monodispersed Sols, Chapter 2, The Colloid Chemistry of Silica, Adv. Chem. Ser. 234, 1994
4. Barth, H.G and S.T. Sun, "Particle Size Analysis", Anal. Chem., 57, 151R-175R, 1985
5. J.G. DosRamos and C.A. Silebi, "Size Analysis of simple and complex mixtures of colloids in the submicrometer ranges using capillary hydrodynamic fractionation", Chapter 19, ACS Symposium Series 472, 1990
6. Iler, R. K., The Chemistry of Silica, John Wiley & Sons, New York, NY, 1979

Figure 1

Fumed Silica Aggregates and Colloidal Silica Particles



a. Fumed Silica SEM



b. Colloidal Silica SEM

Figure 2
"Large Particles" and Gel in Silica Slurries

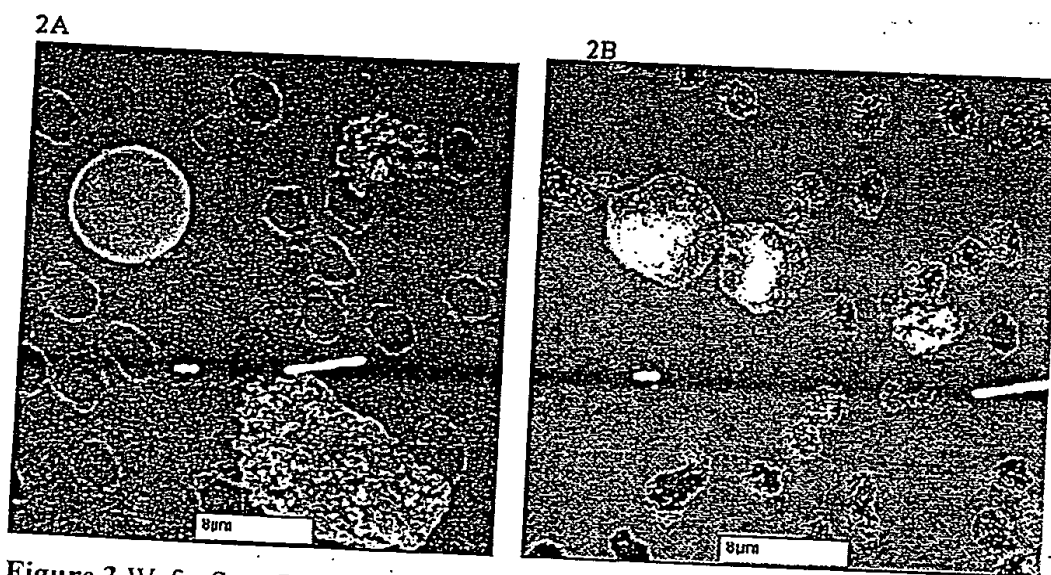


Figure 3 Wafer Scan Result Using Tencor SURFSCAN 6400 for the Wafer Polished with Filtered Slurry

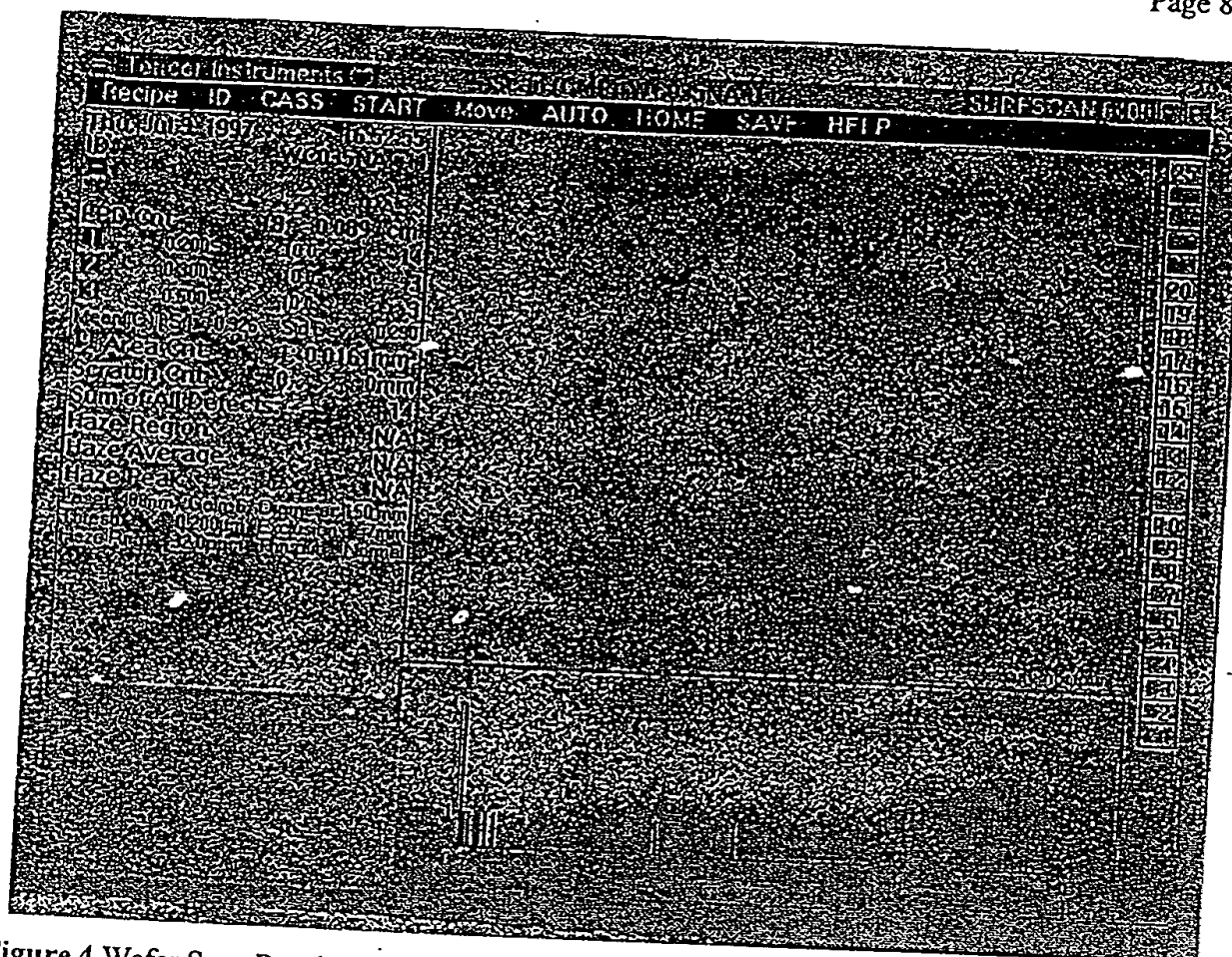


Figure 4 Wafer Scan Result Using Tencor SURFSCAN 6400 for the Wafer Polished with UN-filtered Slurry

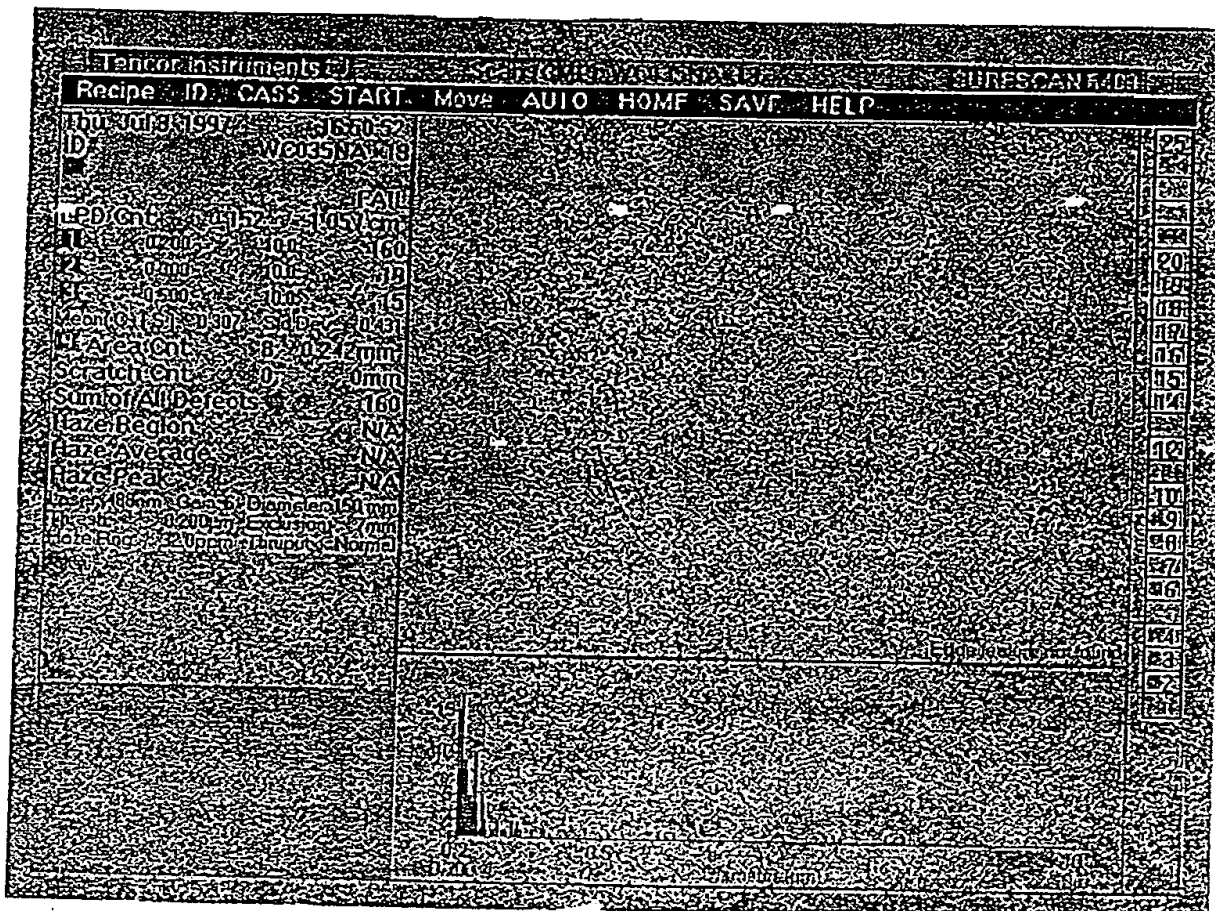


Figure 5

Figure 6

Schematic of Large Particle Counting System

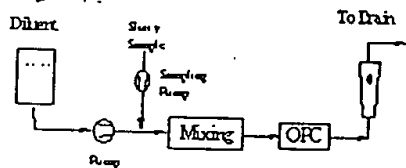


Figure 7
Retention Efficiency of POU
Planargard™ Filters

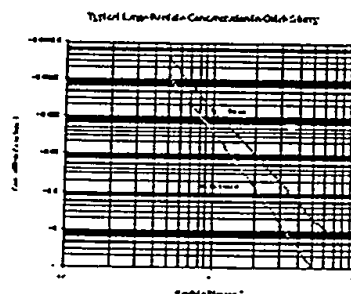


Figure 8
CMP5 Filter Plugging Curve
in Typical Silica Slurry

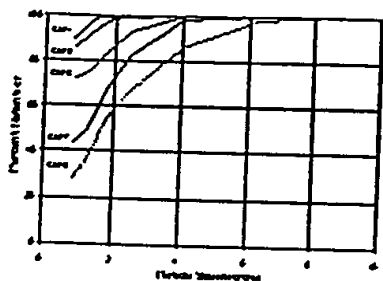


Figure 9
Retention Efficiency and Filter Life

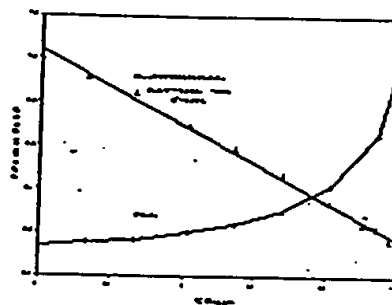


Figure 10
Effect on CMP5 Filter on Slurry Bulk Particle Size Distribution

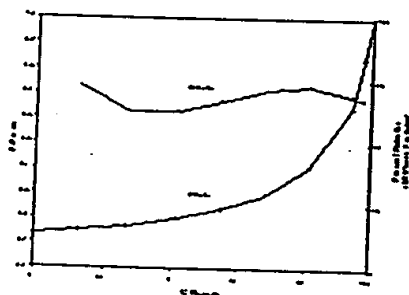


Table I
Effect of Filter Plugging on
Percent Solids and Mean Particle Size in Slurry

	Feed	60% Plugged	95% Plugged
%Solids	12.6	12.4	12.5
DW (nm)	192.3	197.	197.1
DW (nm)	93.3	93.3	94.1

DW: Mean diameter by weight

DN: Mean diameter by number

Table II Field Test Results
Light Point Defect (LPD) Reduction by POU Slurry Filtration

Customer	Slurry	Filters Tested at POU	Normalized LPD Levels
A	Fumed Silica	None	100
		CMP+CMP3	10
B	Colloidal Silica	None	100
		CMP7+CMP5	30
		CMP3+CMP1	9
C	Colloidal Silica	None	100
		CMP3	33

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Lit. No. MA071 Printed in U.S.A. 04/98

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